National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 92

Soda-Lime Glass, Low Boron
(Powder)

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis. It can be used to validate value assignment of in-house reference materials. A unit of SRM 92 consists of a bottle containing approximately 45 g of soda-lime glass powder.

Certified Mass Fraction Values: Certified mass fraction values for SRM 92 are listed in Table 1 [1]. Value assignment categories are based on the definitions of terms and modes used at NIST for certification of chemical reference materials [2]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or taken into account. A certified value is the present best estimate of the true value based on the results of analyses.

Reference Mass Fraction Values: Reference mass fraction values are given in Table 2. Reference values are non-certified values that are the present best estimates of the true values; however, the values do not meet the NIST criteria for certification [2] and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient agreement among multiple analytical methods.

Information Mass Fraction Values: Information mass fraction values are provided in Table 3. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [2]. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 92** is valid indefinitely, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). Accordingly, periodic recalibration or recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of technical measurements for the certification of this SRM was performed by J.R. Sieber of the NIST Chemical Sciences Division. The coordination of the original certification of SRM 92 was performed by H.B. Knowles formerly of the National Bureau of Standards (NBS) Analytical Chemistry Division.

Measurements for value assignment of SRM 92 were performed at NIST by J.R. Sieber of the Chemical Sciences Division. Additional measurements were performed by collaborating laboratories: K. Blanton, R. Embrey, Y. Gao, G. Hay, M. McDonald, S. Robertson, and L.M. Schurter, Owens Corning Science & Technology Center, (Granville, OH); and L. Glaubach, E. Miller, and A.M. Ogura, ALS Minerals Division (North Vancouver, BC, Canada).

Statistical consultation for this SRM was provided by S. Lund of the NIST Statistical Engineering Division.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Robert L. Watters, Jr., Director
Office of Reference Materials

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

To relate analytical determinations to the values on this Certificate of Analysis, a minimum sample quantity of 250 mg is recommended. This minimum mass is sufficient for analyses of the glass matrix composition elements and of all constituent elements using methods with sample preparation by acid dissolution. However, certain measurement techniques may require a greater sample mass. For all constituents, except B_2O_3, the material is used as-received. For determinations of B_2O_3, dry the powder at 105 °C for 1 hour.

Direct analysis using X-ray fluorescence spectrometry (XRF) requires a minimum sample mass of 1.5 g, and the quality of measurements may benefit from using sufficient sample mass to create specimens that are infinitely thick with respect to the measured X-ray of highest energy. In the measurements made at NIST, the highest energy X-rays were Sb K-$L_{2,3}$ at 26.3 keV. At this energy, a sample mass of 3.25 g is required to prepare a 31 mm diameter specimen, for example as a pressed briquette or as loose powder in a liquid cell. In addition, it is recommended to base results for Pb, obtained using XRF measurements, on the average of at least two independent measurements.

Store the material in its tightly-capped, original container in a cool, dry location. Do not return material to the original SRM 92 bottle.

NOTICE TO USERS

NIST encourages the use of its SRMs to establish metrological traceability for the user's measurement results, and NIST strives to maintain the SRM inventory supply. However, NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of SRMs as primary benchmarks for the quality and accuracy of the user's in-house reference materials and working standards. As such, SRMs should be used to validate or otherwise assign values to the more routinely used reference materials in laboratory. When the metrologically traceable values of such reference materials are assigned using this SRM for calibration, the uncertainties assigned to those values must include the uncertainty of the certified value of this SRM, appropriately combined with the uncertainties of the calibration measurements for the in-house reference material. When this SRM is used only to validate the measurement process used to assign a value to an in-house material, inclusion of the uncertainty of the certified value is not appropriate. Comparisons between NIST SRMs and in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

PREPARATION AND ANALYSIS[1]

The material for this SRM was blended and bottled at the NIST facilities (Gaithersburg, MD).

Homogeneity testing was performed at NIST using XRF. The homogeneity of the powder was found to be satisfactory for use with the test methods used in certification. Test methods used by NIST and collaborating laboratories in the development of this SRM are listed in Table 4.

Determinations of B_2O_3 were performed by the following laboratories: NIST (Gaithersburg, MD); Norton Co. (Worcester, MA); Booth, Garrett & Blair (Philadelphia, PA); and Sharp-Schurtz Co. (Lancaster, OH).

Certified Mass Fraction Values: The measurands are the mass fractions of the elements in soda-lime glass listed in Table 1. Metrological traceability is to the SI derived unit for mass fraction (expressed as percent). The values in Table 1 come from fitting a statistical model to the measurements made on the SRM 92 material using multiple test methods. The Bayesian inference paradigm was used for statistical inference [3]. The 95 % expanded uncertainties are equal to one half of the length of the 95 % coverage intervals listed in Table 1, which have been calculated in a manner consistent with the ISO/JCGM Guides [4,5] and express contributions from all recognized sources of uncertainty, including differences between analytical methods, differences among samples, dispersion of values resulting from sample preparation and replicated measurement, preparation and measurement of calibrants, analytical

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[1] Certain commercial equipment, instrumentation, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institutes of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

calibration functions, assay of primary materials, and balance calibration. The listed 95 % coverage intervals are believed to include the true values of the corresponding mass fractions with 95 % probability.

For B_2O_3, the certified value is the best estimate of the true value, on a dry basis, based on the results of a cooperative analytical program. The certified value is not expected to deviate from the true value by more than the stated uncertainty.

Table 1. Certified Mass Fraction Values for SRM 92 Soda-Lime Glass, Low Boron

Constituent	Mass Fraction (%)	95 % Coverage Interval (%)		
Aluminum (Al)	0.706	0.657	to	0.759
Arsenic (As)	0.031	0.022	to	0.041
Calcium (Ca)	5.81	5.36	to	6.30
Iron (Fe)	0.0466	0.0390	to	0.0551
Lead (Pb)	0.0030	0.0025	to	0.0036
Magnesium (Mg)	0.041	0.029	to	0.057
Potassium (K)	0.478	0.438	to	0.521
Silicon (Si)	35.1	31.1	to	39.4
Sodium (Na)	9.6	8.8	to	10.5
Sulfur (S)	0.0164	0.0122	to	0.0222
Boron oxide $(B_2O_3)^{(a)}$	0.70	0.67	to	0.73

[a]The value for B_2O_3 was determined on a dry basis. Dry the material in air for 1 h at 105 °C.

Reference Mass Fraction Values: The measurands are the mass fractions of the elements in soda-lime glass listed in Table 2 as determined by the indicated methods listed in Table 4. Metrological traceability is to the SI derived unit for mass fraction (expressed as percent). The values in Table 2 come from fitting a statistical model to the measurements made on the SRM 92 material using multiple test methods. The Bayesian inference paradigm was used for statistical inference [3]. The 95 % expanded uncertainites are equal to one half of the length of the 95 % coverage intervals listed in Table 2, which have been calculated in a manner consistent with the ISO/JCGM Guides [4,5] and express contributions from all recognized sources of uncertainty, including differences between analytical methods, differences among samples, dispersion of values resulting from sample preparation and replicated measurement, preparation and measurement of calibrants, analytical calibration functions, assay of primary materials, and balance calibration. The listed 95 % coverage intervals are believed to include the true values of the corresponding mass fractions with 95 % probability.

Table 2. Reference Mass Fraction Values for SRM 92 Soda-Lime Glass, Low Boron

Constituent	Mass Fraction (%)	95 % Coverage Interval (%)		
Barium (Ba)	0.12	0.11	to	0.13
Manganese (Mn)	0.002	0.001	to	0.003
Phosphorus (P)	0.002	0.001	to	0.004
Strontium (Sr)	0.005	0.003	to	0.010
Titanium (Ti)	0.008	0.005	to	0.012
Zinc (Zn)	0.22	0.20	to	0.24
Zirconium (Zr)	0.008	0.003	to	0.017

Information Mass Fraction Values: Information values that may be of interest and use to the SRM user are given in Table 3. The values for the listed elements represent the estimated limits of detection of the applied test methods listed in Table 4.

Table 3. Information Values for SRM 92 Soda-Lime Glass, Low Boron

Constituent	Mass Fraction (%)	Constituent	Mass Fraction (%)
Antimony (Sb)	< 0.003	Gallium (Ga)	0.0002
Beryllium (Be)	0.0001	Fluorine (F)	0.02
Bismuth (Bi)	< 0.001	Lithium (Li)	< 0.005
Cadmium (Cd)	0.0007	Mercury (Hg)	45 µg/g
Cerium (Ce)	0.0002	Nickel (Ni)	< 0.0005
Cesium (Cs)	< 0.001	Rubidium (Rb)	0.004
Chromium (Cr)	< 0.002	Selenium (Se)	0.0003
Cobalt (Co)	< 0.0002	Tin (Sn)	< 0.002
Copper (Cu)	0.0013	Vanadium (V)	< 0.0003
		Loss on Ignition at 1000 °C	0.96

Table 4. Analytical Methods Used for SRM 92 Soda-Lime Glass, Low Boron

Constituent	Methods[a]	Constituent	Methods[a]	Constituent	Methods[a]
Aluminum	1, 3, 5	Copper	3, 5	Rubidium	5
Antimony	2, 4, 5	Fluorine	11	Selenium	4, 5
Arsenic	1, 4, 5	Gallium	5	Silicon	1
Barium	1, 3, 5	Iron	1, 3, 5	Sodium	1, 5
Beryllium	3, 5	Lead	1, 2, 4, 5	Strontium	1, 4, 5
Bismuth	3, 5	Lithium	5, 7	Sulfur	1, 5, 6
Boron oxide	10	Magnesium	1, 3, 5	Tin	1, 2, 3, 5
Cadmium	3, 5	Manganese	1, 3, 5	Titanium	1, 3, 5
Calcium	1, 5	Mercury	9	Vanadium	3, 5
Cerium	5	Nickel	3, 5	Zinc	1, 3, 5
Cesium	5	Phosphorus	1, 3, 5	Zirconium	1, 3, 5
Chromium	3, 5	Potassium	1, 5	Loss on Ignition	8
Cobalt	3, 5				

[a] Key to Methods in Table 4:

1. X-ray fluorescence spectrometry after borate fusion
2. X-ray fluorescence spectrometry with standard additions calibration after borate fusion
3. Inductively coupled plasma optical emission spectrometry
4. Graphite furnace atomic absorption spectrometry
5. Inductively coupled plasma mass spectrometry after digestion in a mixture of HNO_3, $HClO_4$, HF, and HCl
6. Combustion with infrared detection
7. Flame atomic absorption spectrometry
8. 2 g powder heated to 1000 °F (537.7 °C) for 1 h, cooled in a desiccator, and weighed
9. Direct mercury analyzer
10. Distillation of boron as methyl borate, complexation, and titration [12]
11. Determination using ion selective electrode after fusion with $LiBO_2$ and acid dissolution

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Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials: telephone (301) 975-2200; fax (301) 948-3730, email srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.